

b. p. 189–194^o (12 mm.); d^{20}_4 1.302; n^{20}_D 1.6110. Attempts to convert this chloride to the desired ethylene by treatment with sodium iodide in dry acetone yielded a mixture of crystalline compounds which could not be separated by repeated recrystallizations. Analysis indicated the product to be a mixture of the two pinacolones,

$\text{C}_6\text{H}_5\text{C}(\text{O})\text{C}(\text{C}_6\text{H}_5)_2$ and $\text{ClC}_6\text{H}_4\text{C}(\text{O})\text{C}(\text{C}_6\text{H}_5)_2$, in spite of precautions to exclude moisture. From 63 g. of *p*-chlorobenzophenone chloride and 70 g. of sodium iodide boiled under reflux in 500 cc. of dry acetone for twelve hours, 45 g. of the mixture was obtained, m. p. 126–145^o. After repeated recrystallization a sample melted at 130–142^o.

Anal. Calcd. for $\text{C}_{26}\text{H}_{18}\text{Cl}_2\text{O}$: C, 74.82; H, 4.35. Found: C, 75.09; H, 4.39.

The desired ethylene was obtained by boiling 5 g. of *p*-chlorobenzophenone chloride in 50 cc. of dry ether with 10 g. of pure zinc dust for one hour. The mixture was filtered, washed with dilute hydrochloric acid, alkali and water and dried over calcium chloride. The ether was evaporated and the solid residue was extracted twice with 20-cc. portions of boiling alcohol. The residue was then crystallized several times from 50 cc. of alcohol to yield 0.5 g. (12%) of *sym-p,p'*-dichlorotetraphenylethylene, m. p. 202–203^o.

Anal. Calcd. for $\text{C}_{26}\text{H}_{18}\text{Cl}_2$: C, 77.81; H, 4.52. Found: C, 77.85; H, 4.56.

When 0.25 g. of the ethylene in 10 cc. of benzene and 10 cc. of absolute alcohol was treated with 1 g. of sodium, a quantitative yield of fine white needles of tetraphenylethane was obtained, m. p. 209^o.³

Anal. Calcd. for $\text{C}_{26}\text{H}_{22}$: C, 93.37; H, 6.63. Found: C, 92.89; H, 6.80.

Reduction of 1.1 g. of the ethylene in 15 cc. of methylcyclohexane with hydrogen at 110 atm. and 100^o using 2 g. of Raney nickel as a catalyst yielded a mixture of products from which 0.3 g. of fine white needles of *p*-chlorotetraphenylethylene was isolated by recrystallization from aqueous acetic acid. The product melted sharply at 168^o; Bergmann and Christiani⁴ report 165–166^o and Norris and Tibbetts,⁵ 162^o.

Anal. Calcd. for $\text{C}_{26}\text{H}_{19}\text{Cl}$: C, 85.11; H, 5.22. Found: C, 85.28; H, 5.43.

(2) Overton¹ reports the boiling point as 192^o (12 mm.); Morgan (*THIS JOURNAL*, **38**, 2100 (1916)) reports 190^o (10–12 mm.); and Ingold and Wilson (*J. Chem. Soc.*, 1493 (1933)) report 191–193^o (13 mm.).

(3) Sagumenny, *Ann.*, **184**, 177 (1877).

(4) Bergmann and Christiani, *J. Chem. Soc.*, 412 (1936).

(5) Norris and Tibbetts, *THIS JOURNAL*, **42**, 2085 (1920).

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dl- and *meso*- γ,γ' -Diphenyl- γ,γ' -suberodilactone

The reduction of β -benzoylpropionic acid with zinc dust in boiling 80–90% acetic acid has been found to yield γ -

phenyl- γ -butyrolactone, m. p. 35–36^o, in 30–40% yield accompanied by the corresponding bimolecular reduction products, the two stereoisomeric γ,γ' -diphenyl- γ,γ' -suberodilactones, in 12–17% yields. For example, 16 g. of β -benzoylpropionic acid in 75 cc. of 80% acetic acid was boiled for three and one-half hours while 25 g. of zinc dust was added in portions. The hot mixture was filtered and the cake of zinc dust was extracted with several portions of hot acetic acid to remove the high-melting dilactone. After several recrystallizations from glacial acetic acid, 1.5 g. (9%) of high melting dilactone was obtained, m. p. 267^o.¹

On cooling the filtered reaction mixture, zinc acetate crystallized. This was removed by filtration and water was added. Crystalline dilactone and oily lactone separated from solution. Filtration yielded 1 g. (6%) of low-melting dilactone. After repeated recrystallization from alcohol, various samples showed a characteristic behavior in that they melted sharply at 165^o to a cloudy semi-liquid which changed to a clear liquid sharply at 175.5^o.

Anal. Calcd. for $\text{C}_{20}\text{H}_{18}\text{O}_4$: C, 74.57; H, 5.63; sapn. eq., 161. Found (267^o): C, 74.55; H, 5.58; sapn. eq.,² 162. Found (165^o): C, 74.34; H, 5.65; sapn. eq.,² 164.

(1) Fieser ("Organic Syntheses," Vol. XV, John Wiley and Sons, Inc., New York, N. Y., 1935, p. 65) has reported the isolation of γ,γ' -diphenyl- γ,γ' -suberodilactone, m. p. 254^o, as a by-product of the Clemmensen reduction of β -benzoylpropionic acid.

(2) Redemann and Lucas, *Ind. Eng. Chem., Anal. Ed.*, **9**, 521 (1937).

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A Dioxanate of Iodine Pentafluoride

Iodine pentafluoride is partially soluble in dry 1,4-dioxane, addition of an excess causing colorless crystals of a dioxanate of formula $\text{IF}_5\cdot\text{C}_4\text{H}_8\text{O}_2$ to precipitate. The crystals start to hydrolyze immediately upon contact with the atmosphere, yielding iodic acid as the only solid residue after complete hydrolysis. They are likewise hydrolyzed over sulfuric acid in a desiccator.

When placed upon a melting point block, the dioxanate melts after an interval of time as low as 84^o, but on immediate contact with the block only at 112^o or above. In all cases melting is accompanied by decomposition and the evolution of hydrogen fluoride and purple iodine fumes.

Analysis was conducted by a modification of the method of Prideaux¹ (samples being weighed by shaking from a weighing bottle). Calculated for $\text{IF}_5\cdot\text{C}_4\text{H}_8\text{O}_2$: IF_5 , 71.6. Found: IF_5 , 69.9.

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